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SOLID-STATE INTERACTION BETWEEN NICKEL AND $YBa_2Cu_3O_x$

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ABSTRACT

The solid-state reaction between nickel and $YBa_2Cu_3O_x$ was studied by hot isostatic pressing of $YBa_2Cu_3O_x$ green compacts in nickel. The microstructure, density and nature of phases present in hot isostically pressed (HIPed) parts were characterized. Resistivity measurements indicated that HIPed parts are not superconducting owing to the loss of oxygen and required an annealing step in oxygen for restoring superconductivity. The diffusion of nickel into $YBa_2Cu_3O_x$ as well as the diffusion of Ba, Cu and Y into nickel was very limited. However, a thin interaction zone consisting of a nickel-rich oxide compound was formed at the Ni-$YBa_2Cu_3O_x$ interface. The presence of such an interaction zone could have a detrimental effect on the thermal stabilization of Ni-$YBa_2Cu_3O_x$ composite wires.

INTRODUCTION

Electrotechnical applications of high T_c superconductors (HTSC) require the fabrication of composite cables made up of many fine HTSC filaments embedded in a metallic matrix. Composite cables are necessary for thermal stabilization (reduction of local temperature rise by Joule heating owing to a current switching into the normal metal by-pass), for minimizing AC losses as well as for a better mechanical behavior. Because the fabrication of $YBa_2Cu_3O_x$ will involve relatively high-temperature heat treatment for its consolidation or oxygen stoichiometry adjustment, it is important to select metals of low reactivity towards this material. In the search for suitable metals, we have studied the solid-state interaction between $YBa_2Cu_3O_7$ and nickel.

MATERIALS AND EXPERIMENTAL PROCEDURES

$YBa_2Cu_3O_x$ was prepared by the reaction of Y_2O_3, CuO and $BaCO_3$ in oxygen at 950°C for 6 hours. The material obtained from this first reaction was crushed and subjected to another identical synthesis step followed by slow cooling and a final annealing step at 500°C for 2 hours. A -150 μm powder was produced by grinding and sieving. Green compacts were then cold isostaticaly pressed and afterwards encapsulated with a surrounding nickel powder bed in copper capsules. Hot isostatic pressing (HIPing) of these capsules was carried out at 940°C and 110 MPa for 4 hours to produce a solid-state interaction between $YBa_2Cu_3O_7$ and the surrounding nickel powder. The interaction zone was studied by energy dispersive X-Ray

analysis (EDX). Resistivity measurements of specimens were carried out by a four point method. The open porosity was computed by measuring the weight gain of specimens infiltrated with p-xylene. The density was obtained from picnometric measurements in p-xylene.

RESULTS AND DISCUSSION

As illustrated in Figure 1, the microstructure of HIPed parts indicates that a significant increase in density occurred. The density of HIPed specimens is 5.95 g/cm^3. No open (interconnected) porosity was present in these parts at this density level. There is some residual porosity whose exact value can be computed from the theoretical density of the material. The theoretical density of $YBa_2Cu_3O_x$ compounds depends upon the value of x. As given by cell parameters (1-2), the theoretical density of $YBa_2Cu_3O_x$ increases from 6.13 g/cm^3 at x=6 to 6.39 g/cm^3 at x=7. Since parts were not superconducting above 77 K after HIPing, the value of x is lower than 6.75 (3). Taking 6.33 g/cm^3 (x=6.75) and 6.13 g/cm^3 at x=6 as the upper and lower bounds of the material density, the residual porosity should be between 3% and 6%.

Metallographic examination of as-HIPed parts revealed that the $YBa_2Cu_3O_x$ is mostly constituted of one phase, except a few isolated grains which were identified as copper oxide. These copper oxide grains likely arose from an incomplete reaction of starting compounds during the synthesis step.

The physical aspect of the Ni-$YBa_2Cu_3O_x$ interface is shown in Figure 2. A relatively thin interaction zone about 10 microns thick was formed at the interface after HIPing at 940°C and 110 MPa for 4 hours. It is also worthy to note that extensive oxidation of nickel occurred during HIPing. Since nickel powder was used instead of bulk nickel, it is possible that oxidation of the nickel powder took place before its consolidation. Even if grain boundary diffusion of oxygen in nickel after its consolidation could also play a role (5), it is most likely that direct oxidation of the surface of nickel particles was the main mechanism. Indeed, no gradient in this grain boundary oxidation was developed (Figure 3) as could be expected from diffusion in the solid.

The EDX analyses of the interface carried out in the interaction zone as well as in $YBa_2Cu_3O_x$ and in nickel near the interface are summarized in Table 1. Only metallic elements were taken into account for computing weight percentages.

The semi-quantitativve analysis of bulk $YBa_2Cu_3O_7$ corresponds well to the theoretical composition of $YBa_2Cu_3O_x$ and there is no significant difference at the 90% confidence level (P=0.1). Even if it was shown that nickel can substitute for copper in the $YBa_2Cu_3O_x$ (6-7), only a small quantity of nickel diffused in $YBa_2Cu_3O_x$ and was observed near the interface. It must be pointed out that the analysis was carried out at a magnification of 6000x with a window of 4 micrometers near the interface.

Table 1 - SEMI-QUANTITATIVE ANALYSES OF METAL ATOMS FOR TYPICAL ZONES AS DENOTED IN FIGURE 3

Element	$YBa_2Cu_3O_x$ (wt%)	Interaction zone (wt%)	Nickel	Theoretical $YBa_2Cu_3O_x$
Y	18,5 (3,5)	0,2 (0,3)#	*	16
Ba	44,3 (3,9)	0,8 (0,5)	*	49,6
Cu	35,1 (4,7)	3,1 (1,8)	*	34,4
Ni	2,5 (0,9)	96,1 (1,4)	100	-

* Non-detected.

\# Values in parentheses are standard deviations.

The interaction zone is constituted almost only of nickel and is limited to about 10 microns. A small percentage of copper (3,1%) was also detected in the interface while the signal of barium and yttrium was not statistically significant. No other elements were detected in nickel which also confirms that the diffusion of copper, barium and yttrium is mainly restricted to a few microns. As-HIPed specimens are non-superconducting above 77 K, and the corresponding X-Ray diffraction pattern (Figure 4a) reveals that the structure is mostly tetragonal indicating that a loss of oxygen occurred during HIPing. This likely led to the oxidation of the nickel powder surrounding the $YBa_2Cu_3O_x$ compound and to the formation of a nickel oxide interaction zone.

The electrical resistivity of as-HIPed specimens is several orders of magnitude larger than that of annealed parts. Typically, the resistivity of as-HIPed parts increases with a decrease in temperature (2.3 ohms at 300 K and 50 ohms at 77 K). When 1 mm thick slices of HIPed parts were heat treated in oxygen for 2 hours at 900°C, cooled at 0.02°C/s to 500°C and annealed at 500°C for 2 hours, an orthorhombic structure was obtained (Figure 4b). As shown in Figure 5, annealed parts in oxygen show a superconductivity onset at about 93 K and zero resistance at about 85 K.

CONCLUSIONS

Hot isostatic pressing was carried out at a temperature of 940°C and 110 MPa for 4 hours to bring about a solid-state interaction between $YBa_2Cu_3O_x$ and nickel. The diffusion of nickel into the $YBa_2Cu_3O_x$ compound was very limited. This interaction zone is about 10 microns thick and is likely constituted of a nickel-rich oxide. Even though the interaction zone is relatively thin, its electrical resistance needs to be assessed to determine if it could be detrimental for the stabilization of such wires. As-HIPed $YBa_2Cu_3O_x$ is tetragonal due to a loss in oxygen and an annealing step is required to restore the superconductivity.

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FIGURE CAPTIONS

Figure 1 - Typical microstructure of as-HIPed $YBa_2Cu_3O_x$

Figure 2 - Physical aspect of the interface formed during HIPing.

Figure 3 - Microstructure showing oxidation of nickel.

Figure 4 - X-Ray diffraction pattern of $YBa_2Cu_3O_x$:
a) As-HIPed; b) HIPed and annealed.

Figure 5 - Temperature-dependence of the resistivity of HIPed and annealed $YBa_2Cu_3O_x$.

TABLE CAPTION

Table I - Semi quantitative analyses of metal atoms for typical zones as denoted in Figure 3.

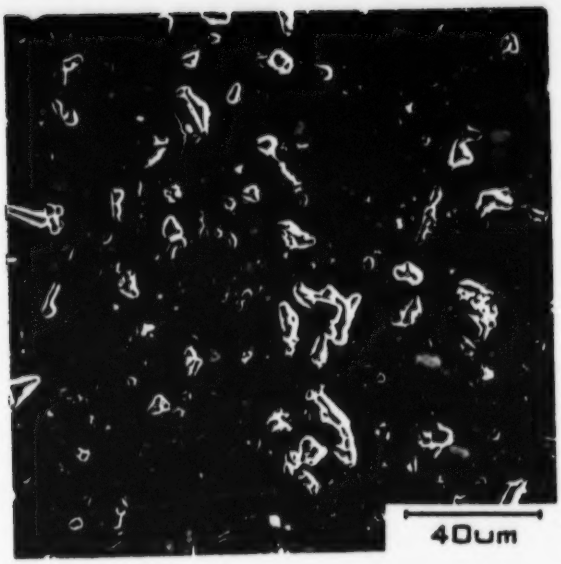


Fig 1

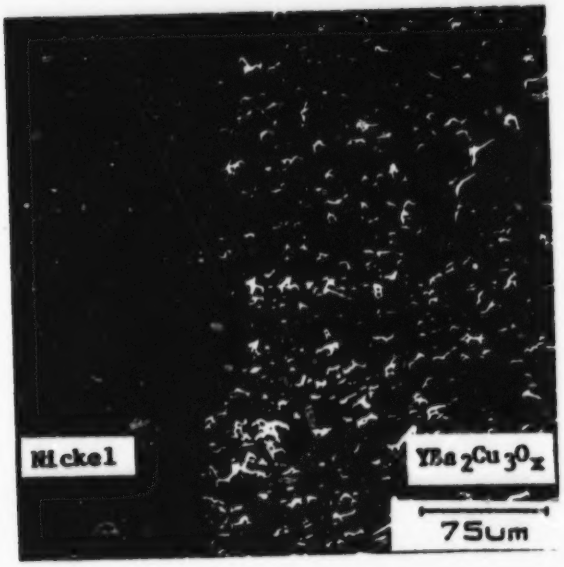


Fig. 2

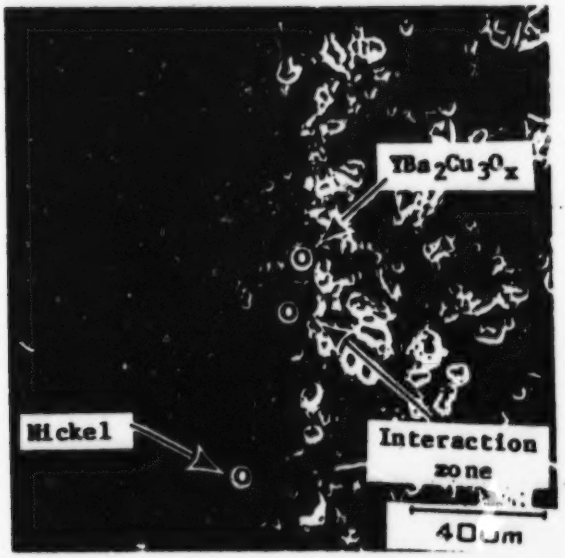
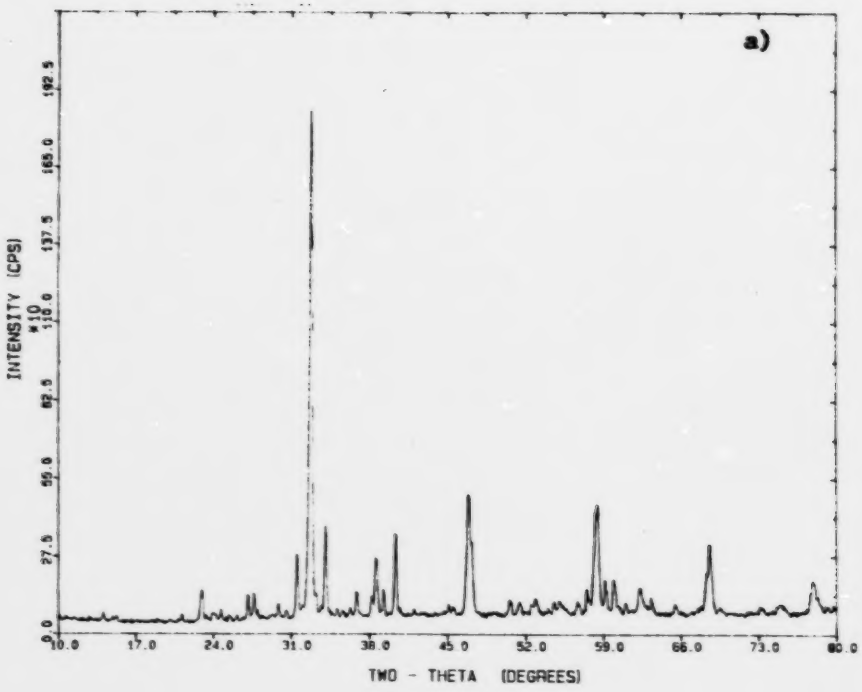


Fig. 3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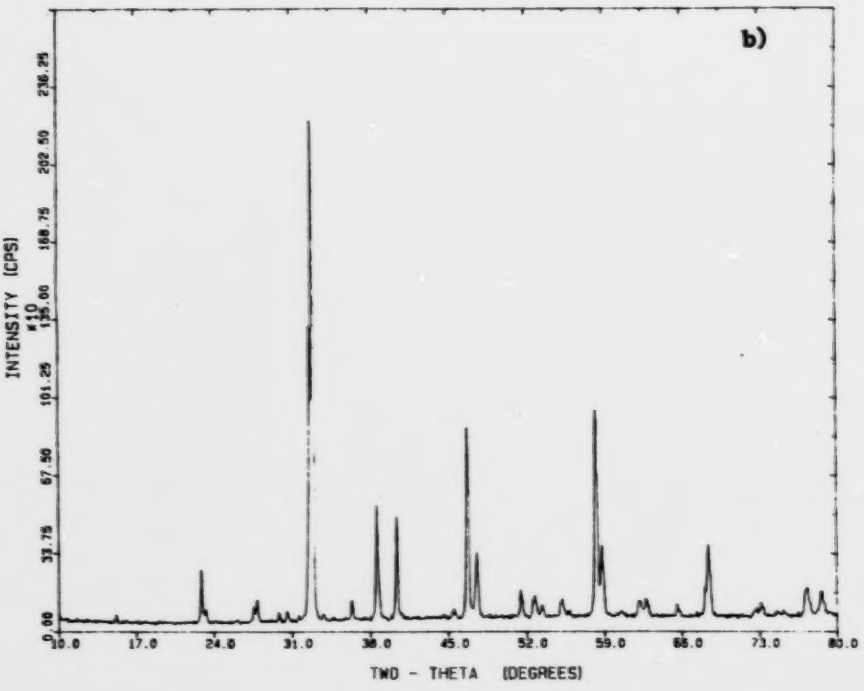
TWO - THETA (DEGREES)

4a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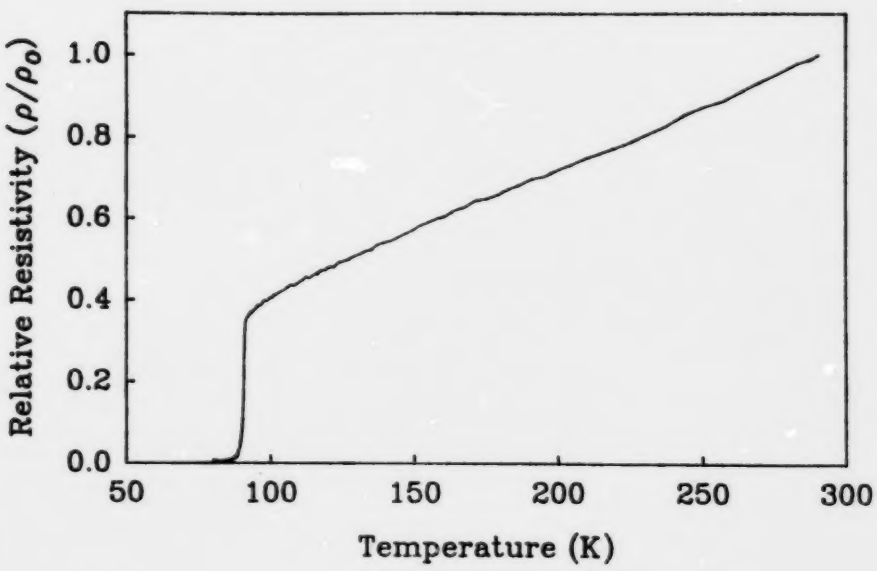
TWO - THETA (DEGREES)

4b


Relative Resistivity (ρ/ρ_o)
1.0
0.8
0.6
0.4
0.2
0.0
50
100
150
200
250
300
Temperature (K)

5